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Title of invention: WATER SOLUBLE CELLULOSE DERIVATIVE HAVING PHOTO-AND HEAT-CROSSLINKABLE PROPERTIES  
Abstract: PURPOSE: To obtain the title derivative free from defects in adhesion and water resistance originated from water soluble properties, causing no sag, etc., and useful for photo-curable coating, ink, etc., by replacing H of hydroxyl group of a specific cellulose ether by a N-methylene acrylamide group. CONSTITUTION: The aimed derivative obtained by replacing H of hydroxyl group of cellulose ether having at least one group among methyl group, 1-4C hydroxyalkyl group and carboxymethyl group as a substituent group and being 0.3-2.0 in sum of average degree of substitution (DS) per unit glucose ring of substituent group thereof by N-methylene acrylamide (-CH<sub>2</sub>-NH-CO-CH=C H<sub>2</sub>). COPYRIGHT: (C)1990, JPO&Japio

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## [WHAT IS CLAIMED IS]

## [Claim 1]

A water-soluble cellulose derivative; comprising: The methyl basis, hydroxyalkyl groups of carbon equation 1 to 4, a total of substitution degree, at a minimum, one is comprised as a substituent and to average unit glucose *kanto rino* of they substituent in a thing of the carboxymethyl basis (DS) are the light bridging characteristics that a hydrogen atom of a hydroxyl group of the cellulose ethers which are less than 2.0 substitutes in the N- methylene acrylic amide basis ( $-\text{CH}_2 - \text{NH-CO-CH} = \text{CH}_2$ ) more than 0.3, and it is and heat bridging characteristics.

## [DETAILED DESCRIPTION OF THE INVENTION]

(a field of industrial application) while having the cellulose derivative that the present invention is new, properties of matter same as particularly existing water-soluble cellulose ethers, there are not adhesive power due to a water-soluble thing and a defect in water resistance, and an extremely useful solution cellulose derivative is related to in industry, besides, to have both light bridging characteristics and heat bridging characteristics. (prior art:) binders in producing are wide, and, as for the water-soluble cellulose ethers represented with a methyl cellulose, hydroxyethyl cellulose, hydroxypropyl cellulose, a hydroxypropylmethylcellulose, carboxymethylcellulose conventionally, is employed an extruding body of emulsion system paint, kamitokozai, zonenai such as various glue, a skin of film forming agent, earth-water preservation agent and fluidity improvement agent, ceramic system of cement mortar and cement system, but, there is a problem of some unresolved breakdown in these. (a problem to be solved by the invention), for example, the film which it dries, and is formed becomes poor in water resistance after the painting when water-soluble cellulose ethers are used to paint pro-emulsion, kamitokozai, glue. In addition, Because viscosity of the painting thing does not become so high till quantity included water equivalent evaporates by these uses, it is in the way, and *dare* is produced, and long time is needed for expression of adhesive power to be directed to, and work efficiency is a problem not to increase. On the other hand, A product of soluble polymerization characteristics is known to an organic solvent substituted for cellulose ethers of 2.0-2.9 water indissolubility or the N- methylene acrylic amide basis ( $-\text{CH}_2 - \text{NH-CO-CH} = \text{CH}_2$ ) of substitution degree 0.05-0.5 to average a hydrogen atom of a hydroxyl group of ester (Japanese Patent Laid-Open No. 60-144301 bulletin), and a unit glucose *kanto rino* ave. substitution degree (it is assumed merely DS as follows) of a substituent is not known to the thing which solved this problem in water-soluble cellulose ethers. Thus, Adhesive power develops in a short time without object of the invention producing *dare* due to a water-soluble thing in spite of being durability in properties of matter same as existing water-soluble cellulose ethers, after water resistance improves the new solution cellulose derivative which, even more particularly, have both light bridging characteristics and heat bridging characteristics is going to be provided. (a means for solving problem) scholars of present invention solve a problem in water-soluble cellulose ethers such as the above, the superior characteristic is kept alive, and to use effectively, zealously, as a result of study, in a thing of the methyl basis, hydroxyalkyl groups of carbon equation 1 to 4, the carboxymethyl basis, at a minimum, one is comprised as a substituent and a total of DS of they substituent finds that a solution cellulose derivative having the light bridging characteristics that a hydrogen atom of a hydroxyl group of the cellulose ethers which are less than 2.0 more than 0.3 substitutes in the N- methylene acrylic amide basis ( $-\text{CH}_2 - \text{NH-CO-CH} = \text{CH}_2$ ), and it is and heat bridging characteristics is equal to the object, if it is completed with the present invention, it was reached. As follows, The details of the present invention are explained. For the methyl basis to compose a water-soluble cellulose derivative, hydroxyalkyl groups of carbon equation 1 to 4 of the present invention, solution cellulose ethers comprising one as a substituent at least in a thing of the carboxymethyl basis, a methyl cellulose, hydroxyethyl cellulose, hydroxypropyl cellulose, a hydroxypropylmethylcellulose, carboxymethylcellulose are illustrated. It is less than 2.0, and, as for the present invention, a total of DS of a substituent has the structure that a hydrogen atom of glucose *kan* is substituted for in the N- methylene acrylic amide basis ( $-\text{CH}_2 - \text{NH-CO-CH} = \text{CH}_2$ ) more than 0.3 in this kind of water-soluble cellulose ethers. This is less than 0.3, and, as for a total of DS being limited to a thing of less than 2.0 more than 0.3 here, solution is lost, in addition, then a cost becomes high, and economy is lost more than 2.0, the reason is because improvement of a characteristic to balance with it cannot be anticipated. About DS of the N- methylene acrylic amide basis, there is not limitation in particular, but, it is more than 0.05 for achievement of object of the invention. This situation is preferable. Production of a water-soluble cellulose derivative of the present invention assumes cellulose ethers comprising DS a starting material to the public, is done by reacting N- *mechiroruakuriruamido* to be able to be elaborate. The cellulose ethers, N- *mechiroruakuriruamido*, an acid catalyst, polymerization prohibition agent were dissolved in these mixture solvents or after, besides, this method scattered water or alcohol, an organic solvent such as acetone, though N- *mechiroruakuriruamido* of quantity of indispensability responds, temperature and time to need are given, and it is reacted, is achieved by it is poured, and doing postprocessing appropriately, but, it is exchanged with this and, cellulose, a methyl black ride, ethylene oxide, propylene oxide, mono these ether makes N- *mechiroruakuriruamido* coexist to an agent in an agent and a manufacturing process of cellulose ethers to react ether such as chloroacetic acid, generation reaction and N- *mechiroruakuriruamido* of cellulose ethers are preferable as a method to make travel reaction at the same time. To an acid catalyst used by a method of the former, there are phosphoric acid, hydrochloric acid, Para toluene sulfonic acid, an ammonium chloride, citric acid, tartaric acid, hydroquinone, hydroquinone methyl ether, Para benzoquinone are given to polymerization prohibition agent again, these consider the dissolution characteristics to the solvent which employs respectively, and choice can be employed appropriately. For example, a drying step is passed through, and, for a postprocessing process, it is done volatilization such as solvent by a method to get a product refined by the rough product which it is able to have been removed is washed in a suitable solvent, and drying by reaction liquid after the reaction end. In addition, On the occasion of production of this water-soluble cellulose derivative, previously described reaction can be done with drying by heat-treating mixtures such as solvent, cellulose ethers, N- *mechiroruakuriruamido*, an acid catalyst, polymerization prohibition agent by vacuum drying, oven drying, drum drying, a suitable dry method such as spraying drying. In addition, When a product dissolves in reaction liquid, it makes precipitate an object product it is *hin\*bai* and and reaction liquid and a solvent getting mixed uniformly are doped to reaction liquid as a precipitant for a product, after having separated from by filtering, centrifugation, it can be done with a product by operating the refinement. On the other hand, When it dispersed without a product dissolving in reaction liquid, it was filtered directly or after having separated from by centrifugation, pass, and refinement operation can be manufactured. One part of hydrogen atom of hydroxyl groups included in cellulose ethers substituted N- *mechiroruakuriruamido* for the chemical structure of a water-soluble cellulose derivative with the present invention. Because of this, While this water-soluble cellulose derivative is basic, and having properties of matter same as cellulose ethers (by presence of a polymerization initiator, ultraviolet rays, light irradiation such as an electron beam and heat-treatment, polymerization or bridging reaction travels), *\*yu* does a function of the N- methylene acrylic amide basis. After a water-soluble cellulose derivative of the present invention having such a property used a polymerization initiator together in the emulsion paint which was a use of conventional water-soluble cellulose ethers, kamitokozai such as an overcoat of feeling

fever paper, glue, and having done coating, light is irradiated or it is heat-treated, adhesive power develops instantly again, and work efficiency improves without *dare* producing bridging reaction produces between cellulose derivatives, and to solidify, and water resistance improves, and problems in conventional solution cellulose ethers can dissolve entirely. In addition, Because when light hardening from before-related resin (a light hardening-related monomers, light hardening-related oligomer) is used together, water getting wet characteristics, blocking characteristics resistance, *koshino* strength can be given to light hardening characteristics resin, now light hardening characteristics resin can expect used light hardening characteristics paint, ink, expansion of demand in a field such as glue. In addition, In light hardening used here-related resin, two - echiruheshirukureto, 1,6- hexanediols diacrylate, light hardening characteristics monomer such as trimethylol propane bird acrylate, polyester acrylate, epoxy acrylate, polyurethane acrylate, light hardening characteristics oligomer such as polyeter acrylate are given , to these resin and a polymerization initiator used together, hyperoxidation benzoyl, benzyl, biasechiru, *azobisuisobuchironitoriru* are given. (an example) concrete embodiment of the present invention is explained by an example as follows, but, the present invention is not a thing limited to this example. It is 1 to a reactor of feather having roots to agitate example 1 While enter, and agitating *no* ethyl acetate, hydroquinone 0.2g, 85% phosphoric acid 0.4g, *N-mechiroruakuriruamido* 75g were increased, and it was dissolved. In next, *hidoroshikipuopiruseru* (DS:) 1.75) 100g are increased, it was responded at reaction temperature 50 degrees Celsius for six hours. The coarse material which is able to have been filtered after cooling with a reactant is cast into heated water of 90 degrees Celsius, it dried, and, after washing, refined goods were got. DS of each substituent of these refined goods was as follows. The *hidoroshikipuopiru* basis : The DS =1.73 N- methylene acrylic amide basis: It is 1 in 2. DS =0.45 example beakers Entered, and 85% phosphoric acid 2.0g, hydroquinone methyl ether 6g, *N-mechiroruakuriruamido* 300g were increased to be able to be elaborate, and the third grade *no* butanol was dissolved. While agitating, hydroxypropyl cellulose 1,000g used with a precedent were increased, and it was mixed. A prepared slurry is filtered, filtering operation was canceled at a point in time that *ro* liquid of 800g exhausted. A left cake is not assigned to an aluminum container evenly, after having done, it could be to a dryer of 50 degrees Celsius, and it heated for ten hours. Coarse material provided in next is cast into heated water of 90 degrees Celsius, it dried, and, after washing, it was assumed refined goods. DS of these refined goods was as follows. The *hidoroshikipuopiru* basis : The DS =1.74 N- methylene acrylic amide basis: Replacing with hydroxypropyl cellulose in 3. DS =0.31 example example 1, as for the others using a methyl cellulose (DS =1.81), DS of provided refined goods was as follows when operation in much the same way as example 1 was done. The methyl basis : The DS =1.80 N- methylene acrylic amide basis: Replacing with hydroxypropyl cellulose in 4. DS =0.31 example example 1, as for the others using carboxymethylcellulose (DS =0.65), DS of provided refined goods was as follows when operation in much the same way as example 1 was done. The carboxymethyl basis : The DS =0.63 N-methylene acrylic amide basis: After casting did the thing which dissolved cellulose derivative 100g and *biasechiru* 3g provided with 5. DS =0.37 example example 1 to 4s in pure water 400g on a glass board, it is left unattended at temperature 20 degrees Celsius, a film of half dehydration of 50 depthwise  $\mu\text{m}$  was got. A high-pressure mercury-vapor lamp of lamp input 80W/cm was used for this film of run speed 5m/, and ten times of ultraviolet rays were irradiated. A provided film is left at decompression bottom room temperature, the water which remained behind within a film was removed. After a dipping did this film 5g to pure water of 500g for one hour, quantity of dissolved film is measured, indissolubility demanded a rate by the next expression, and it was done with an aim of bridging characteristics and solvent characteristics resistance.

$$\text{不溶化率 (\%)} = \frac{\text{5 - 溶解したフィルム分 (g)}}{5} \times 100$$

The result was shown in a separate table. After casting did the thing which dissolved cellulose derivative 100g and *azobisuisobuchironitoriru* 3g provided with 6. example example 1 to 4s in pure water 400g on a glass board, it was heat-treated at 100 degrees Celsius for five hours, and a film of 100 depthwise  $\mu\text{m}$  was got. Processing same as a precedent is made on this film, and indissolubility demands a rate, the result was written jointly

セルロース誘導体	フィルムの不溶化率	
	実施例 5	実施例 6
実施例 1 で得られたもの	97	98
// 2 //	97	97
// 3 //	98	96
// 4 //	96	95

at the next separate table.

(an effect of the invention) after a water-soluble cellulose derivative with the present invention used a polymerization initiator together in the emulsion paint which was a use of conventional water-soluble cellulose ethers, kamitokozaï such as an overcoat of feeling fever paper, glue, and having done coating, light is irradiated or it is heat-treated, adhesive power develops instantly again, and work efficiency improves without *dare* producing bridging reaction produces between cellulose derivatives, and to solidify, and water resistance improves, and problems in conventional solution cellulose ethers can dissolve entirely. In addition, Because when light hardening from before-related resin (a light hardening-related monomers, light hardening-related oligomer) is used together, water getting wet characteristics, blocking characteristics resistance, *koshino* strength can be given to light hardening characteristics resin, now light hardening characteristics resin can expect used light hardening characteristics paint, ink, expansion of demand in a field such as glue.